(21) Application No. 13045/75 (22) Filed 27 March 1975

(31) Convention Application No.

2 415 159 (32) Filed 29 March 1974 in

(33) Fed. Rep. of Germany (DT)

(44) Complete Specification published 29 June 1977

(51) INT. CL.² C11D 11/02

(52) Index at acceptance

C5D 6A3 6A5B 6A5C 6A5D2 6A5E 6A9 6B12G1 6C6 6D

(72) Inventors NIKOLAUS PECHTOLD JOACHIM BUCHWALD

(54) ALKANESULPHONATE SPRAY PRODUCT

(71) We, HOECHST AKTIENGESELL-SCHAFT, a Body Corporate organised under the laws of the Federal Republic of Germany of 6230 Frankfurt/Main 80 Germany, 5 do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a spray product containing an alkali metal alkanesulphonate as surfactant and to a process for its

preparation. It has been proposed to prepare detergent powders by spray drying mixtures of active detergent substances, e.g. soaps, alkarylsulphonates, alkenesulphonates and alkanesulphonates, together with other pre-20 dominantly inorganic additives as carriers. Whereas spray drying of mixtures of the first three above-mentioned detergent substances and inorganic additives generally yields free-flowing, pourable powders which 25 do not cake together on storing, the products of spray drying mixtures of alkanesulphonates and inorganic additives generally have poor storage properties if the detergent content is high. Since alkanesul-30 phonates are hygroscopic, spray products produced therefrom with a high content of alkanesulphonates tend to cake or even become sticky as a result of water absorption from the atmosphere or from the inorganic 35 additives which contain water of crystallisation. For this reason alkanesulphonates have hitherto been used satisfactorily only

and bath additives.

40 There have been numerous attempts to overcome the problem of the poor storage properties of alkanesulphonates and to facilitate the preparation of spray products containing alkanesulphonates by adding 40 small quantities of so-called "anti-caking agents" to the spray mixture. Thus benzene-

in solution e.g. in liquid cleaning materials

sulphonates, toluenesulphonates and magnesium silicate have for example been proposed as "anti-caking agents". In addition, high-melting, finely divided powders which 50 are not hygroscopic have been added to the spray-dried products. Such powders act purely mechanically by coating the individual granules of the spray product, thus preventing them from sticking to one 55 another. It is also possible to reduce caking on storage by greatly increasing the proportion of carrier to detergent but this is commercially undesirable in that the detergent strength of the powder is reduced. 60

According to the present invention we now present a free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) 65 at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15%, preferably at least 20% by weight referred to the combined 70 weight of carrier and alkanesulphonate and and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of alkanesulphonate.

The spray-dried powders according to the 75 invention are in general free-flowing, nonhygroscopic and thus non-caking. They may be prepared by spray-drying an aqueous composition containing A) a carrier comprising at least one salt of an inorganic or 80 organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15%, preferably at 85 least 20% by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of al- 90 kanesulphonate at a temperature from 100 to 350°C, preferably from 120 to 320°C,



which process constitutes a further feature of the invention.

The alkanesulphonate component of the spray-dried powders according to the inven-5 tion preferably comprises alkanesulphonates containing from 12 to 18 carbon atoms in the alkyl group and the alkali metal is preferably sodium or potassium.

The silica is conveniently added to the 10 aqueous spray composition in the form of a dispersion having a bulk weight of from

60 to 400g per litre.

The carrier generally should have a melting point of at least 150°C. Salts of non-15 corrosive acids are preferred, particularly their sodium salts such as sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate and sodium citrate. Small quantities of sodium chloride 20 may also be contained in the spray mixture, for example as impurity introduced into the above-named sodium salts during their preparation. Other suitable carrier salts include for example potassium and am-25 monium sulphate, potassium carbonate and a potassium and an ammonium polyphosphate. Mixtures of carrier salts may also be used. The carrier is preferably soluble in water at 100°C in an amount of at least 30 30% by weight.

The spray-dried powder may if desired contain other additives conventionally used in washing powders, for example optical

brighteners, bleaches and soaps.

The process of preparing the spray-dried powder according to the invention may be effected in a conventional spray plant. Thus for example the hot aqueous spray composition may be atomised by passage 40 through a nozzle and brought into contact with a flow or counterflow of hot gases. The aqueous composition is preferably as concentrated as possible for the spray-drying method. Water contained in the spray 45 composition evaporates and a spray product is obtained which consists of approximately spherical, non-caking particles. The bulk weight and water content of the spray product may be adjusted by selecting appropri-50 ate spray conditions, the spray temperature being particularly important.

The spray-dried powders according to the invention may be used as components of surfactant compositions optionally in ad-55 mixture with the aforementioned additional additives. They may also be used as lubricants or carriers is cosmetic preparations. It is also possible to use the process according to the invention for a one-step pre-60 paration of commercial detergent com-

positions.

The following Examples serve to illustrate the preparation of spray-dried powders according to the invention.

65 Example 1

A 35% aqueous spray solution containing a mixture of sodium sulphate and sodium alkanesulphonates (containing, on average, 15 carbon atoms in the alkyl group) in a weight ratio of 1.5:1 is prepared. 4% by 70 weight (based on the weight of alkanesulphonates) of a fine dispersion of silica with a bulk weight of 100g per litre is dispersed in the aqueous spray solution. The mixture is then sprayed at a temperature between 75 315 and 340°C. A white, free-flowing, non hygroscopic spray-dried powder with a water content of about 7% by weight and a bulk weight of 300 to 400g per litre and which does not cake on storage is obtained. 80 Example 2

A 32% aqueous spray solution containing a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate 85 in a weight ratio of alkylsulphonates to tripolyphosphate of 1:3 is prepared. 0.375% by weight (based on the weight of alkanesulphonates) of a fine silica dispersion with a bulk weight of 100g per litre is dispersed 90 therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, nonhygroscopic spray-dried powder with a bulk weight of 300 to 400g per litre and a water content of 10% by weight and which does 95 not cake on storage.

Example 3

A 45% aqueous spray solution containing a mixture of sodium alkanesulphonates containing predominantly alkanesulphonates 100 with 12 to 15 carbon atoms, sodium sulphate and sodium tripolyphosphate in a weight ratio of alkanesulphonates to sulphate to tripolyphosphate of 1:2:2 is prepared. 1.1% by weight (based on the weight 105 of alkanesulphonates) of a fine silica dispersion with a bulk weight of 200g per litre is dispersed therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, non-hygroscopic spray-dried 110 powder with a bulk weight of 180 to 250g per litre and a water content of 7% by weight and which does not cake on storage. Example 4

A spray-dried powder is prepared ana- 115 logously to Example 1 except that the aqueous spray mixture is sprayed at a temperature of 290 to 300°C. A white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 300 to 400g 120 per litre and a water content of about 8% by weight and which does not cake on storage is obtained.

Comparison Example A

Spray products are prepared analogously to 125 Examples 1 to 2 but omitting the silica dispersion. The products are white and initially free-flowing but cake on storage.

Comparison Example B

A 33% aqueous spray solution containing 130

a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate in a weight ratio of alkanesulphonates to tri-5 polyphosphate of 2:15 and containing no silica dispersion is sprayed analogously to Example 1 to yield a white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per

10 litre and a water content of 15% by weight. Comparison Example C A spray-dried powder is prepared analogously to comparison Example B except 15 that tetrasodium pyrophosphate is used in the place of sodium tripolyphosphate. A

white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per litre and a water 20 content of 15% by weight is obtained.

WHAT WE CLAIM IS:-

1. A free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali 25 metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15% by weight referred to the combined weight of 30 carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by weight referred to the weight of alkanesulphonate.

2. A spray-dried powder as claimed in claim 1 wherein the content of silica is from 35 is from 0.3 to 4% by weight referred to

the weight of alkanesulphonate.

3. A spray-dried powder as claimed in claim 1 or claim 2 wherein the carrier has

a melting point of at least 150°C.

4. A spray-dried powder as claimed in any of the preceding claims wherein the carrier is sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate, sodium citrate, potassium carbon-45 ate, a potassium polyphosphate, potassium sulphate, ammonium sulphate or an ammonium polyphosphate.

5. A spray-dried powder as claimed in claim 3 or claim 4 wherein the carrier is :50 soluble in water at 100°C in an amount of

at least 30% by weight.

6. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises an :55 alkanesulphonate containing from 12 to 18 carbon atoms.

7. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises a 60 sodium or potassium alkanesulphonate.

8. A spray-dried powder as claimed in any of the preceding claims wherein the content of alkanesulphonate is at least 20% by weight referred to the combined weight 65 of carrier and alkanesulphonate.

9. A spray-dried powder as claimed in any of the preceding claims which additionally contains at least one further ingredient selected from optical brightening agent, bleaches and soaps.

10. A spray-dried powder as defined in claim I substantially as herein disclosed.

11. A spray-dried powder as defined in claim 1 substantially as herein disclosed.

with particular reference to Example 1 to 3. 75 12. A spray-dried powder as defined in claim 1 substantially as herein described with particular reference to Example 1 to 3.

13. A process for the preparation of a free-flowing spray-dried powder containing 80 an alkali metal alkanesulphonate as surfactant which comprises spray-drying at a temperature of from 100 to 350°C an aqueous composition containing A) a carrier comprising at least one salt of an in- 85 organic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15% 90 by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by weight referred to the weight of alkanesulphonate.

14. A process as claimed in claim 13 wherein the composition is spray dried at a temperature of from 120 to 320°C.

15. A process as claimed in claim 13 or claim 14 wherein the content of silica in 100 the composition is from 0.3 to 4% by

16. A process as claimed in any of claims 13 to 15 wherein the silica is introduced into the aqueous composition in the 105 form of a fine dispersion having a bulk weight of 60 to 400g per litre.

17. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described.

18. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described with reference to Examples 1 to 4.

19. A process for the preparation of 115 spray-dried powder according to claim 13 substantially as herein described with refer-

ence to Examples 1 to 3.

20. A free-flowing spray-dried powder whenever prepared by a process as claimed 120 in any of claims 13 to 18.

21. A surfactant composition comprising a free-flowing spray-dried powder as claimed in any of claims 1 to 12 and 20.

For the Applicants FRANK B. DEHN & CO. Chartered Patent Agents. Imperial House. 15-19 Kingsway London, WC2B 6UZ.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1977. Published at the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.